FIRST QUARTERLY REPORT

Ending

July 27, 1961

PEM FOR PRODUCTION OF FLUORINATED BARIUM TITANATE CAPACITORS FOR OPERATION TO 200°C

CONTRACT NO. DA-36-039-SC-85955
U. S. ARMY SIGNAL SUPPLY AGENCY
PHILADELPHIA, PENNSYLVANIA

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CDE
CORNELL-DUBILIER
ELECTRIC CORPORATION
CERAMIC DIVISION · NEW BEDFORD, MASSACHUSETTS

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By

CORNELL-DUBILIER ELECTRIC CORP.

Ceramic Division

New Bedford, Mass.

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1. ABSTRACT

1.1 The manufacture of ceramic capacitors suitable for operation to 200°C represents a new area of interest. The problems associated with this type of capacitor require specialized equipment, assembly techniques and materials. Specialized equipment has been and is being constructed to provide rapid testing and evaluation of fluorinated capacitors. Life test equipment permitting external monitoring of capacitors during test is close to completion. In addition, a high temperature chamber has been constructed for rapid evaluation of dielectric constant, dissipation factor and insulation resistance over a wide temperature range.

1.2 A study was made of the reports on degradation and fluorination of ceramic dielectrics as reported by Linden Laboratories, Inc. A sound approach to the problem of manufacture of fluorinated ceramic capacitors which would permit maximum use of present production facilities was established. A two fire approach is planned, the first representing a binder burn off and partial sintering to be followed by a fluorination-maturing firing. It is believed that this would provide maximum control of the fluorination treatment. A temporary tunnel kiln has been assembled. A pusher mechanism has also been constructed to provide accurate soaking periods from 30 minutes to two hours. The fluoride atmosphere will be provided by introduction of volatile fluorides on a regular schedule.

2. PURPOSE

2.1 The purpose of this project is to establish the capability to fluorinate 16,000 capacitor discs per eight hour shift and to manufacture 4000 capacitors each of four types in accordance with Signal Corps Technical Requirements SCS-37 dated 9 March, 1959 and amendment No. 1 dated 29 November, 1960. This requirement defines the quality and testing program for 200°C fluorinated ceramic capacitors as listed below.

2.1.1 CK63 barium titanate capacitors rated at 10,000 mmf. +-20%, 500 VDC at 85°C and 250 VDC at 200°C using barium titanate made with West German barium carbonate.

2.1.2 CK63 barium titanate capacitors rated at 10,000 mmf. +-20%, 500 VDC at 85°C and 250 VDC at 200°C using barium titanate made with domestic barium carbonate.

2.1.3 Barium titanate capacitors, maximum diameter 0.39 inches rated at 10,000 mmf. +-20%, 50 VDC at 85°C and 25 VDC at 200°C using barium titanate made with West German barium carbonate.

2.1.4 Barium titanate capacitors, maximum diameter 0.39 inches rated at 10,000 mmf. +-20%, 50 VDC at 85°C and 25 VDC at 200°C using barium titanate made with domestic barium carbonate.

3. NARRATIVE AND DATA

3.1 INTRODUCTION

The problems involved in the manufacture of fluorinated ceramic capacitors suitable for operation to 200°C require an intensive evaluation of many factors. Some of the more pertinent items are as follows:

3.1.1 Dielectric composition best suited for maximum fluorination benefit.

3.1.2 Reproducible and controlled method of fluorination.

3.1.3 Assembly and encapsulation materials suitable for operation at 200°C.

3.2 SPECIAL TEST FACILITIES

3.2.1 The work accomplished in this first quarter can be best described as construction and preparation for solution of the first two items. The test equipment and facilities will serve as tools for quality control and experimental investigations. In view of this approach, the life test oven and associated fixtures have been so designed as to permit external monitoring of each capacitor on test. It is believed that this will allow maximum flexibility and speed for evaluating a large variety of compositions and fluorination trials simultaneously.

3.2.2 The most notable change in the ceramic dielectric after fluorination appears to be its increased insulation resistance at elevated temperatures. This phenomena plus the change in dielectric

constant, dissipation factor and color should provide a good means of evaluating control of the fluorination firing. A special high temperature test chamber has been constructed for this purpose.

3.3 FLUORINATION TECHNIQUE

The techniques of fluorination and the resultant improvement to the dielectric has been extensively investigated by Linden Laboratories. An examination of the reports on this matter and discussions with the personnel involved in this work have helped formulate the approach to be followed.

3.3.1 A two fire operation was decided upon. The unfired capacitor discs would be fired in standard production kilns. This eliminates the problem of fire out of organic binders in the fluorination firing. The degree of firing would be determined by the results of the fluorination treatment. The other advantages of using prefired discs are smaller size, greater flexibility of loading and that fluorination equipment can be simplified and more easily controlled. The second firing or fluorination treatment will be performed in a continuous pusher type tunnel kiln. An experimental kiln for this purpose, along with a mechanical pusher, has been constructed. The kiln has a 20 inch hot zone, 3 inches wide and 1 1/8 inches high. The over-all length of the tunnel is approximately 56 inches. The heating elements are isolated

from the work zone by nitride bonded silicon carbide plates 1/2 inch thick. The mechanical pusher has a 4 speed range giving soaking periods from 30 minutes to 2 hours. This kiln will be used for experimental firings leading to the construction of the final kiln.

3.3.2 The fluoride atmosphere will be provided by additions of volatile fluorides on a regular schedule. This method of fluoride additions has been successfully tried.

4. CONCLUSION

Based on the results of fluorination treatment by Linden Laboratories, Inc., an ambitious program of dielectric improvement has been sponsored by the U. S. Signal Corps. The fulfillment of this contract will result in ceramic capacitors capable of extended life at 200°C. To this end, a program which would permit a thorough investigation of compositions and fluoride treatment has been planned. Special equipment and kilns have been constructed for this purpose.

5. PROGRAM FOR NEXT QUARTER

5.1 INTRODUCTION

The establishment of a reproducible fluorination cycle with barium titanate will establish the firing and charging period of the fluorination kiln. This data can be correlated with that obtained by Linden Laboratories. When satisfactory operation of the fluorination kiln is completed, all effort will be directed towards development of an optimum composition.

5.2. COMPOSITION

As was noted by Linden Laboratories, all compositions are not equally improved by fluorination and considerable work is required to arrive at a composition that will give maximum reliability at 200°C. A series of binary compositions with barium titanate will be evaluated to broaden knowledge of fluorination and also will serve as an aid in selecting the best capacitor formulation. The final compositions should have the following properties after fluorination:

5.2.1 Dielectric constant of approximately 5000

5.2.2 Aging rate not to exceed 1.6% per decade

5.2.3 Withstand a voltage stress of 30 volts per mill for 2000 hours at 200°C

5.2.4 Exhibit high insulation resistance throughout temperature involved.

5.2.5 Withstand assembly and encapsulation techniques required.

5.3 ASSEMBLY

In keeping with the need for a high temperature capacitor, conventional assembly techniques using high temperature materials will be evaluated. The continued improvement of organic resins indicate that this approach might be satisfactory. If this approach does not provide a high quality capacitor capable of meeting all test requirements, the use of silver wire leads and glass encapsulation will be investigated.

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Born April 2, 1920

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